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METHOD FOR DIRECT DETERMINATION OF ALUMINA IN REFRACTORY MATERIALS

(First Revision)

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Indian Standard

METHOD FOR DIRECT DETERMINATION OF ALUMINA IN REFRACTORY MATERIALS

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 27 June 1979, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 This standard was first published in 1959 as a tentative standard. In this revision, the earlier methods have been replaced by an EDTA method for direct determination of alumina in refractory materials.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard covers the method for direct determination of alumina in high alumino refractories and alumino-silicates containing low (less than 2 percent) lime and magnesia.

2. SAMPLING AND PREPARATION OF SAMPLE

2.1 The material shall be sampled in accordance with the procedure laid down in IS : 1528 (Part VII)-1974†.

2.2 Laboratory Sample — The samples shall be crushed in any type of crusher with hardened tool steel faces with due precautions to prevent contamination of the sample with steel particles from the sampling equipment during crushing and grinding. After quartering, 20 g of the sample

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*Rules for rounding off numerical values (*revised*).

†Methods of sampling and physical tests for refractory materials: Part VII Method of sampling and criteria for conformity (*first revision*).

shall be carefully gone over with a magnet to remove extraneous iron particles. It shall then be ground in an agate mortar so that it passes completely through IS Sieve 15 (aperture 149 microns, *see* IS : 460-1962*).

2.3 Test Sample — It is prepared for the purpose of chemical analysis by drying a sufficient quantity of the laboratory sample in a glass weighing bottle in an air oven at 105 to 110°C.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be used.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. OUTLINE OF THE METHOD

4.1 The sample is decomposed with hydrofluoric and sulphuric acids and the ignited residue is fused with potassium pyrosulphate or potassium bisulphate and extracted with acid. Aluminium is separated as sodium aluminate after treatment with sodium hydroxide and complexed quantitatively with EDTA by boiling with an excess of the reagent. The excess EDTA is back titrated with standard zinc solution at about *p*H 5·2 using xylenol orange indicator. The aluminium EDTA complex on boiling with ammonium fluoride or sodium fluoride liberates EDTA which when titrated with standard zinc solution gives the amount of aluminium present in the solution.

5. REAGENTS

5.1 Dilute Sulphuric Acid — 1 : 1 (*v/v*). Add 10 ml of concentrated sulphuric acid (rd = 1·84) cautiously with stirring to 100 ml of water and cool.

5.2 Hydrofluoric Acid — 40 percent.

5.3 Potassium Bisulphate/Pyrosulphate — solid.

5.4 Dilute Hydrochloric Acid — 1 : 1, 1 : 4 and 1 : 10 (*v/v*).

5.5 Sodium Hydroxide Solution — 10 percent (*m/v*).

5.6 Sodium Carbonate — solid.

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*Specification for test sieves (*revised*).

†Specification for water for general laboratory use (*second revision*).

5.7 EDTA Solution (0·025 M) — Dissolve 9·31 g of disodium salt of ethylene-diamine tetracetic acid, dihydrate ($C_{10}H_{14}N_2Na_2O_8, 2H_2O$) in water and dilute to 1 litre.

5.8 Methyl Orange — Dissolve 0·05 g of methyl orange in 100 ml of water.

5.9 Dilute Ammonia — 1 : 5 (*v/v*).

5.10 Acetate Buffer (*p*H 5·2) — Dissolve 21·5 g of sodium acetate ($CH_3COONa, 3H_2O$) in 300 ml of water containing 2 ml of acetic acid and dilute to one litre.

5.11 Standard Aluminium Solution (1·0 mg Al_2O_3/ml) — Weigh 0·529 3 g of aluminium metal (99·9 percent purity) into a beaker containing 40 ml of dilute hydrochloric acid (1 : 1). Heat the beaker over a water-bath. Transfer the solution to 1 000-ml volumetric flask and make up to the mark.

5.12 Standard Zinc Solution (0·025 M) — Dissolve 5·484 5 g of zinc acetate [$Zn(CH_3COO)_2, 2H_2O$] in 50 ml of water, 0·3 ml of glacial acetic acid and 25 ml of acetate buffer (*see* **5.10**) and dilute to one litre in a measuring flask. Find out the equivalent Al_2O_3 in g/ml from the procedure given in **6.3** taking 20 ml of standard aluminium solution (*see* **5.11**).

5.13 Xylenol Orange Indicator — Dissolve 0·25 g of xylenol orange in 100 ml of water containing two drops of dilute hydrochloric acid (1 : 1).

5.14 Ammonium Fluoride Solution (20 Percent) — Dissolve 20 g of ammonium fluoride in 100 ml of water. Add 2 drops of xylenol orange (*see* **5.13**) and then dilute hydrochloric acid (1 : 10) dropwise till the colour changes to yellow.

6. PROCEDURE

6.1 Solution of the Sample — Weigh accurately 0·2 to 0·5 g of well agated and dried (at 105°C) sample of the finely ground test sample in a platinum dish. Moisten the sample with 25 ml of water. Add 3 to 5 ml of the dilute sulphuric acid and 15 ml of hydrofluoric acid and cover the dish loosely with the platinum lid. Allow to evaporate slowly to fuming on the hot-plate or sand bath in a fume cupboard, taking care to avoid spurting. Repeat the process with 5 ml of hydrofluoric acid and 5 ml of water. Cool, add 2 ml of the dilute sulphuric acid and evaporate as before. Heat the dry residue until fumes of sulphur trioxide cease to be evolved. Raise the temperature to 1 000°C and ignite for five minutes. Cool and fuse the residue with a little potassium pyrosulphate or potassium bisulphate, dissolve the melt in dilute hydrochloric acid (1 : 4) (*see* Note).

NOTE — After dissolving the melt in dilute hydrochloric acid, if the solution is not clear, filter and wash the residue 2 to 3 times with a little dilute hydrochloric acid (1 : 10). Ignite the residue in a platinum crucible and then fuse with about 1 g of sodium carbonate and borax (2 : 1) mixture. Extract the melt with dilute hydrochloric acid (1 : 4) and add to the filtrate.

6.2 Make the volume to about 75 ml and nearly neutralize with sodium hydroxide solution. Slowly and while swirling constantly pour the solution into a 250-ml volumetric flask containing 100 ml of sodium hydroxide solution to which has been added 1 g of sodium carbonate (*see* Note). Warm on a water bath for one hour and cool to room temperature. Dilute with water to the mark, mix well and let stand for a few minutes. Filter off the precipitate on a filter paper that has been previously washed with sodium hydroxide solution, catching the filtrate in a beaker (rinse the beaker first with a few ml of the same filtrate). Transfer a suitable aliquot from the filtrate to a 250-ml conical flask. Add one drop of methyl orange and then dilute hydrochloric acid (1 : 1) dropwise until the colour changes from yellow to red.

NOTE — The sodium carbonate is used to precipitate calcium and magnesium along with iron and titanium.

6.3 Add sufficient EDTA solution to provide an excess of at least 5 ml over the expected amount of alumina. Add dilute ammonia dropwise until the colour changes from red to yellow. Add 5 ml of acetate buffer solution and about 25 ml of water and heat to boiling for 5 minutes. Cool, add 5 ml of buffer solution and titrate with standard zinc solution using 3 to 4 drops of xylenol orange indicator. The end point is indicated by a clear change from yellow to deep orange colour. Add 10 ml of ammonium fluoride solution and heat to boiling for 5 minutes. Cool, add 5 ml of acetate buffer solution and titrate the liberated EDTA with standard zinc solution.

7. CALCULATION

$$\text{Alumina, percent} = \frac{A \times B \times 100}{C}$$

where

A = volume in ml of standard zinc solution required for titration of liberated EDTA,

B = equivalent Al_2O_3 in g/ml of standard zinc solution, and

C = mass in g of the sample represented by the aliquot taken.

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